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(57) Abstract

	<p>(57) Abstract</p> <p>A process for the production of artificial leather which comprises coating or impregnating a fibrous base material for the production of artificial leather with an aqueous emulsion of a polymer exhibiting elasticity in a solid state and subjecting the resulting base material to both wet heat (utilizing heat of water vapor) and irradiation with microwave to solidify the emulsion. As this process can dispense with an organic solvent, it is friendly to the environment, is reduced in the migration as compared with the conventional dry-heat solidification processes using hot air, and can give an artificial leather which has distinct voids (noncontact areas) indispensable to the feeling of artificial leather between the fibers of the base material and the polymer incorporated into the material, is excellent in air permeability by virtue of innumerable extremely fine pores formed when the water vapor generating in the solidification passes therethrough, and contains pores also in the polymer solidified in the base material. This process can dispense with also the shrinking step which has been essential in the prior art, thereby simplifying the production process, and can be shortened in the processing time so as to enhance the production efficiency.</p>	
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Disclosure

Manufacturing method of artificial leather

Technological field

This invention pertains to the manufacturing method of artificial leather.

Technological background

The suggested improved technology for the manufacture of artificial leather with natural leather touch and quality pertains to the ultrafine fibers, which is the base for the fiber base material, to the polymer for coating or impregnation of the fiber material, and to the pretreatment for coating or impregnation of the fiber base material. For dilution the polymer, dimethylamide (DMF) (sic, Translator's Note: If it were DMF then the compound should be "dimethyl formamide"), which is harmful to human, was used.

This organic solvent, beside the deterioration of the workplace environment in processing plant, it requires hot water rinsing, water rinsing therefore it uses a large quantity of water, and when discard the rinsed water it creates water pollution and air pollution. To prevent this, the organic solvent in the wastewater or waste gas was recycled and the recycled solvent must processed. These processed cost an enormous labor and cost and it is inconvenient.

In the organic solvent using method, as shown in Fig. 7, the method comprises a fiber base material shrinking process; a polyvinyl alcohol (PVA) impregnation process (In this process, the fiber base material was treated with polyvinyl alcohol to prevent the adhesion of polymer on the fiber in the fiber base material.); a drying process; a polymer dilution process; a polymer coating or impregnation process; a polymer condensation process; a hot water rinse and water rinsing

process; a squeezing process; a drying process; and coiling process to obtain the product. This method has too many processes and is inconvenient.

To alleviate these problems, for example, a water base emulsion of polyurethane resin solution was diluted to a given concentration with water and the resulting solution was impregnated in non-woven fabric and others. After these processes, the treated material was processed according to the process using an organic solvent, and it is conceivable that it was then processed through drying and curing processes to bond the polyurethane on the fiber base material.

Since an organic solvent was not used in this method, unlike the process using an organic solvent, the associated process such as hot water and water rinsing process can be eliminated in the method. Hence, a product can be obtained through a fiber base material shrinking process, emulsified polymer coating process, pre-drying process, drying process, and polymer bonding process. It is beneficial because of the lesser processes than one using an organic solvent.

However, after trying the method following problems were encountered.

One of the problem was that during the initial drying period, after impregnating a fiber base material with water-emulsion polyurethane resin solution and during the evaporation of water, the polyurethane resin solid mass moves in the fiber base material, a migration phenomenon. due to this phenomenon, water evaporates from inside of the fiber base material and the resin mass will migrates toward front side and backside of the fiber base material, and this results in thin polyurethane resin in the inside of the fiber base material. This may be the main cause of the uncomfortable touch or feel.

Also, after drying with dry heat, a dry heat bonding was carried out with hot air (120-150°C). For example, when a water emulsion polyurethane resin was used, then the bonded polyurethane resin content in the fiber base material was less than 10%, as compared to 25% to 50% of that when a polyurethane resin in an organic solvent was used.

The reason is that after a conventional dry-heat drying/dry heat bonding process to increase the bonding mass weight, then the polyurethane resin adheres on the fibers of the fiber base material and becomes hard as shown on the electron micrograph 5 which shows

the artificial leather structure. And in the subsequent processing, for example, microfiber forming technology of sea island type fiber, or dyeing process will become difficult.

Thus prepared artificial leather was dyed by an solution flow dyeing apparatus or vat steam dyeing apparatus by leaching processing or dyeing processing but in this mode the solution flow dyeing apparatus operation becomes difficult and spots will be formed when leaching process was carried out, which becomes the cause of dye spotting in the dyeing process. Also, in the vat steam dyeing, the touching of artificial leather is unavoidable and this becomes the cause of the smudging, which is unacceptable.

By the invention, a water base emulsion of polyurethane, which is pollution-free, an artificial leather with a soft and close to natural leather can be manufactured, which is the target of this invention.

Development of Invention

In the patent application, a fiber base material for artificial leather manufacture was coated or impregnated with a water emulsion of polyurethane, which becomes elastic after bonding, then the polymer solution in the fiber base material was bonded on the fiber base material by wet heat and microwave, which is the characteristics.

when an artificial leather was manufactured by the method, the processing time was significantly reduced and thus manufacture artificial leather is very soft and has elasticity as compared those of the artificial leather made using the conventional water base emulsion polymer aqueous solution. Also, during the wet-heat bonding process, the shrinkage of fibers confirmed and the migration was insignificant as compared with the conventional hot air drying in dry-heat bonding process. Also, the polymer impregnated between the fibers in the fiber base material has void (non-contact area) was formed, which is the necessary conditions for the nice feeling of an artificial leather. Moreover, the steam generated during the bonding process forms many tiny pores, which provides permeability. Additionally, the conventionally necessary shrinking process can be eliminated.

Explanation of Figures

Fig. 1 is an electron micrograph in lieu of a figure for the artificial leather structure manufactured by invention method in Example 1.

Fig. 2 is an electron micrograph in lieu of a figure for the artificial

leather structure manufactured in Comparison 1.

Fig. 3 is an electron micrograph in lieu of a figure for the artificial leather structure manufactured by other example of the invention method.

Fig. 4 is an electron micrograph in lieu of a figure for fiber splitting and fractionating treatment after example in Fig. 3.

Fig. 5 is an electron micrograph in lieu of a figure for the artificial leather structure manufactured by conventional method.

Fig. 6 is a work flow sheet of the invention method.

Fig. 7 is a work flow sheet of conventional method.

Fig. 8 is a work flow sheet of conventional method.

Most acceptable form of invention example.

In order to describe the invention in detail, the invention will be described with figures.

For the fiber base material for artificial leather, nonwoven, woven, and knit material were used.

Among them, the preferred fiber base material, which provide most natural leather feel in the final products, is nonwoven fabrics made from polyamide (nylon fiber) or polyester base fibers.

Especially, the fiber base material containing copolymerized polyester base fiber can be easily split and fractionate by sodium hydroxide, and using the copolymerized polyester base fiber for fiber base material is one possibility in obtaining a soft artificial leather. Also, to improve the quality, it is preferred that the fiber must have thermal shrinking property and easily shrinkable fiber must contain. To the above fiber base material was coated or impregnated with a water base emulsion state polymer (water base elastic polymer, hereafter), which exhibits elasticity when bonded. The coating or impregnation of the fiber base material with the water base emulsion polymer was carried out splitting and fractionating of fibers or before splitting and fractionating of the fiber base material.

In coating method, there are direct coating, reverse coating, gravure coating, spraying etc., when the fiber base material was impregnated with water base elastic polymer then the fiber base material was squeezed by squeeze rollers to control the amount of water base elastic polymer in the fiber base material.

Also, an emulsified polyurethane resin in water base dispersing

agent was used for the water base elastic polymer.

After coating or impregnating of fiber base material with water base elastic polymer, the water base elastic polymer was bonded by using steam heat (wet heat) and microwave heat in a bonding apparatus (steamer hereafter) to bond by wet heat.

When fiber splitting and fiber fractionating with sodium hydroxide after bonding of fiber base material containing copolymerized polyester base fibers, a preferred emulsion is obtained by emulsification with an aliphatic isocyanate base polyether type polyurethane resin emulsion, in detail, urethane polymer with isocyanate end group, which has durability and alkali resistance in polyurethane resin, and then the chain lengthening with polyamine to obtain the preferred aliphatic isocyanate base polyether type polyurethane.

Also, if the fiber base material has shrinking property then shrinking phenomenon occurred at the same when wet heat bonding was carried out. This eliminates the shrinking process which took place before the wet heat bonding process using conventional water base elastic polymer, which is beneficial.

It is preferred that the wet heat bonding was carried out in the atmosphere at 100% of volumetric relative humidity (saturated steam).

However, it is possible to do by steam heating other than saturated steam.

By microwave heating, the temperature of the polyurethane, the water base elastic polymer in the fiber base material, can be raised from the core, and this processing time is shorter than when water base elastic polymer was bonded on fiber based material by wet heat only as shown in Comparison 1.

Moreover, by irradiating with microwave, the bonding of polyurethane, water base elastic polymer, becomes even, and many voids (micro-pores) were formed in the bonded polyurethane resin. After wet heat bonding, the leather-like sheet had soft touch as compared with those of bonded (curing) by a conventional dry bonding method. Subsequently, this wet leather-like sheet was dyed in wet state.

The dyeing process is vat steam method and liquid flow dyeing method, and the latter method, in which dyeing process took place in a vat while rubbing, was preferred. in respect to the feel and touch.

The dyed leather-like sheet was treated a finishing process, buffing etc., when necessary, to obtain an artificial leather. Moreover, the artificial leather obtained by this method creates many micropores in the polyurethane resin during the bonding process by steam from the evaporation occurred during the bonding process, and this provides an excellent permeability.

In the above discussion, the leather-like sheet immediately after wet heat bonding becomes a final product without drying of the sheet, but when buffing was provided, the leather-like sheet after the wet heat bonding was dyed after the wet heat bonding process, and then buffed.

In the above descriptions, for a water base elastic polymer, a water base emulsion of polyurethane resin dispersed in water base dispersing agent was used, but it is not limited that.

Example 1

Polyamide fiber, 0.2 denier single fiber weight after fractionation, 50% and polyester 50% mixture raw cotton was needle punched to 1.4 mm thickness and 300 g/m² unit weight nonwoven fabric (fiber base material).

The above nonwoven fabric was immersed/impregnated in a water base elastic polymer, 10% aqueous solution of water base polyurethane emulsion (solid content 40%), and then squeezed with squeeze rollers to control the aqueous solution content in the above nonwoven fabric. The water content was 160.3%.

Without drying, the bonding of the polymer solution was carried out by wet heating and microwave heating at following conditions.

Wet heat bonding conditions

Steam temperature	100-110°C
Fabrication time	1 minute
Microwave powder output	10 kW.

The moisture evaporation rate after the wet heat bonding was 31.6%

The hardness and the shrinkage are listed in Table 1 and Table 2, respectively.

Subsequently, the above leather-like sheet obtained by the above method was processed a liquid flowing dying apparatus without drying and the process was carried out smoothly and after the sheet treatment there was no clogging of nozzle and the processing was

smooth.

The sheet was then dyed in brown by following conditions.

Kayanol Brown RX (Nippon Seiyaku Product)	2% o.w.f.
Ionet SAD	0.5% o.w.f.
Sodium acetate	0.5 g/L
Acetic acid	0.3 cc/L
Temperature	98°C
Time	60 minutes.

After drying, the unit area weight before and after dying was measured and the polyurethane weight loss was 3%.

The leather-like sheet was dried and the leather-like sheet was buffed

with sandpaper to form a suede like artificial leather.

Thus obtained artificial leather was softer and more elastic without the

shrinkage process as compared to conventional artificial leather.

Shrinkage was observed during the wet heat bonding process time and

migration was much less than conventionally dry heat bonding with hot air,

and significant voids (noncontacting) between the fibers and elastic polymer, the polyurethane resin, which is necessary condition for feel and touch for artificial leather, as shown in electron micrograph 1 in lieu of a figure (without fiber splitting and fractionation). Also, it was confirmed that the elastic polymer became porous.

Furthermore, as shown in Table 4, the adhesion weight of polyurethane resin was high.

Comparison 1

The nonwoven fabric prepared according to in Example 1 procedure was immersed/impregnated in water base polyurethane emulsion in water used in Example 1, squeezed with squeezing rollers to control the weight of the aqueous solution in the above nonwoven fabric, and then wet heat bonded in the steamer used in Example 1. The water content was 156.4%.

without drying, wet heat bonding process was carried out according to following conditions.

Steam temperature
Processing time

100-110°C
4 minutes

The moisture evaporation rate after the wet heat bonding process was 30.9%. The hardness and shrinkage are listed in Table 1 and Table 2, respectively.

The leather-like sheet obtained by the above method was dyed according to Example 1 procedure without drying.

Thus obtained artificial leather took longer wet heat bonding time than Example 1. There are voids (noncontact part) between the fibers and the elastic polymer, the polyurethane resin, which is necessary for the feel and touch of artificial leather but the voids were smaller than those in Example 1. After the bonding process, the touch is somewhat repulsive feeling and the porosity of the bonded polyurethane was unacceptable and the soft touch was not there.

Comparison 2

The same nonwoven fabric in Example 1 was subjected to shrinkage processing at the same temperature and humidity of the wet heat bonding process in comparison 1 procedure. The shrinkage is listed in Table 2. The shrinkage was similar to that occurred in temperature bonding (sic) of Comparison 1.

The shrinkage treated nonwoven fabric was immersed/impregnated in a polyurethane emulsion in water under the same conditions as in Comparison 1 (same as those of Example 1). The water content was 150.3%.

Subsequently, the nonwoven fabric was transported by pin tender and dried and cured (hot air dry heating method).

Drying	120-150°C	3 minutes
Curing	150°C	3 minutes.

The hardness of thus processed leather-like sheet is listed in Table 1 and it was very hard when compared that with Example 1 specimen. When it was dyed through liquid flow dying apparatus, it was very difficult to feed into the apparatus due to the high bulk, even if the nozzle diameter was enlarged; dying was impossible.

Example 2

Shrinkable 3 denier polyester raw cotton was used, and formed to a nonwoven fabric (fiber base material) of 1.3 mm thickness and

255 g/m² unit weight by needle punching method.

The above nonwoven fabric was immersed/impregnated in water base polyurethane aqueous solution according to Example 1 procedure, and squeezed by squeezing roller to control the content of aqueous solution in the nonwoven fabric. The water content was 145.9%.

Subsequently, the above nonwoven fabric was wet heat bonded at the same conditions as in Example 1.

The water evaporation rate was 37.4% during the wet heat bonding process. The hardness and the shrinkage are listed in Table 1 and Table 2, respectively.

The hardnesses immediately after wet heat bonding and shrinkage are listed in Table 2, and it was softer than untreated nonwoven fabric.

The leather-like sheet obtained by the above method was fed to liquid flow dyeing apparatus without drying and the operation was smoother than that of leather-like sheet obtained in Example 1. After the treatment, the nozzle did not clogged up and the operation ran smoothly.

The leather-like sheet was dyed by following conditions.

Dianix Brown 3B-FS (Mitsubishi-Hoechst) 2% o.w.f.

Acetic acid 0.2 cc/L

Sunsolt SN-30 (Nikka Kagaku) 0.25 g/L

Temperature 130°C

Time 30 minutes

After drying, the unit area weight before and after dyeing was measured, and the polyurethane resin loss was 5%.

The dried leather-like sheet was buffed by sandpaper and a velour-like artificial leather was obtained.

Thus obtained artificial leather without shrinking process was softer and more elastic than conventional artificial leather. The sheet had sufficient shrinkage during the wet heat bonding of polyurethane resin. It had less migration than conventional dry bonding process as shown in electron micrograph 3 (without fiber splitting and fractionation), in lieu of a figure, and voids (noncontact parts) between the fibers and the elastic polymer, polyurethane resin, were observed, and the voids are the necessary to provide nice fell and touch of

artificial leather.

Electron micrograph 4, in lieu of a figure, of after fiber splitting and fractionation, is the same magnification as that of electron micrograph 3.

Comparison 3

The same nonwoven fabric in Example 2 was processed for shrinkage at the same wet heat conditions, temperature and humidity, in Example 2. The shrinkage is listed in Table 2, and the shrinkage was similar to that from wet heat shrinkage in Example 2.

The shrinkage treated nonwoven fabric was immersed/impregnated in polyurethane emulsion aqueous solution under the same conditions in Example 1. The water content was 145.9%.

Subsequently, the nonwoven fabric was transported by pin tender and then dried and cured (in hot air dry heating method) under same conditions in Comparison 1.

The hardness of thus processed leather-like sheet is listed in Table 4, and it was harder than that in Example 1. The feeding of the leather-like sheet to a liquid flow dyeing apparatus was difficult due to the high bulk, even if the nozzle was enlarged. Dyeing was impossible. Example 3

The above examples used 10% concentration aqueous solution of water base polyurethane resin, but the processing time can be further shortened by increasing the resin concentration and an addition of dielectric substance in the aqueous solution.

For example, the same nonwoven fabric used in Example 1 was used, and three aqueous solutions of 10%, 15%, and 20% concentration were prepared by using the same water base emulsion (40% solid content) used in Example 1, and then a dielectric substance, which readily converts microwave energy to heat, was added to the aqueous solutions. To the aqueous solutions, the above nonwoven fabric was immersed/impregnated and squeezed by squeezing rollers to control the aqueous solution content in the nonwoven fabric, and then processed in a steamer according to Example 1.

Steam temperature	100-110°C
Processing time	15 seconds
Microwave power output	10 kW.

The results are listed in Table 3, and the water-base polyurethane emulsion concentration (40% solid content included) of 15% had above 96% polyurethane resin bonding.

In Table 3, the one treated in water base polyurethane emulsion concentration (includes 40% solid content) of 20% aqueous solution was dried by dry heat drying process and then the processed according to the above experimental conditions for the comparison of the data.

The dielectric substances are titanium oxide, barium titanate, silica, magnesium carbonate, diethylene glycol etc.

Also, by increasing the resin concentration, the yield was increased by shortening of the bonding processing time. At the same time, after the immersion/impregnation process, the water content can also be reduced after squeezing process. This prevents the dripping of resin in the nonwoven fabric was reduced and this enabled to manufacture an artificial leather with homogeneous resin bonding.

Table 1

Base material	Specimen	Length (mm)	Width (mm)
Polyamide fiber 50%	Unprocessed	77	50
Polyester fiber 50%			
Polyamide fiber 50%	Ex. 1, Microwave	119	106
Polyester fiber 50%	wet heat bonding		
Polyamide fiber 50%	Comp. 1	117	105
Polyester fiber 50%	Wet heat bonding		
Polyamide fiber 50%	Comp. 2	>141	>141
Polyester fiber 50%	Dry heat bonding		
Polyester fiber	Unprocessed	108	118
Polyester fiber	Ex. 2 Microwave	68	56
	Wet heat bonding		
Polyester fiber	Comp. 3	>114	>114
	Dry heat bonding		

Table 2

Base material	Specimen	Length (%)	Width (%)
Polyamide fiber 50%	Ex. 1, Microwave	3.1	2.9
Polyester fiber 50%	wet heat bonding		
Polyamide fiber 50%	Comp. 1	3.2	2.8
Polyester fiber 50%	Wet heat bonding		
Polyamide fiber 50%	Comp. 1	3.6	3.2
Polyester fiber 50%	Dry heat bonding		
Polyester fiber	Ex. 2 Microwave	16.8	19.4
	Wet heat bonding		
Polyester fiber	Comp. 3	17.7	20.7
	Dry heat bonding		

Table 3

Water emulsion concentration	10% (25% aqueous solution)		15% (37.5% aqueous soln.)		20% (50% aqueous solution)		Dry heat
Conditions	M	S	M	S	M	S	
Base material Wt, kg	55.47 57.47		55.34	56.68	55.39 57.80	57.12	
Wt. after immersion in aq. emulsion, kg	144.412	147.11	143.67	147.85	142.16 150.80	174.40	
Pick up, %	160.30	155.9	159.6	160.8	156.6 159.87	205.3	
Polyurethane solid, (Calculated value, kg)	8.89	8.96	13.24	13.67	17.35 18.48	23.45	
Wt. after water rinse, kg	62.85	63.29	68.12	62.23	72.37	73.68	58.9
Polyurethane solid, kg	7.38	5.82	12.78	5.55			
16.98 16.56 1.1Polyurethane solid, %	11.74	9.19	18.76	8.91	23.46 1.86	22.47	
Polyurethane bonding rate, %	83.0	64.9	96.5 65.17		97.75 5.95	70.61	

Remarks: M designates microwave and wet heat, S designates Wet heat only.

By the invention artificial leather was obtained without using an organic solvent and this method is environment friendly and the invention leather is softer and more elastic than conventional artificial leather. Also, shrinkage occurred during the wet heat bonding process, and it has less migration than in the conventional hot air dry heat bonding process. There are clear voids (noncontact part) between the fibers and the polymer impregnated in the fiber base material, and the voids is necessary condition for feel and touch of artificial leather. The steam generated during the bonding process created many fine pores in the polymer, which makes the leather permeable, and porosity in the polymer bonded on the fiber base material was also confirmed. The shrinking process was eliminated, which was a necessary process in conventional manufacturing method, and the manufacturing method was simplified by omitting the shrinking process. Moreover, the processing time was reduced and the production yield was increased.

Possibility of Industrial Applications

The artificial leather obtained by the invention method can be used for gentlemen's shoe tops, women's shoe tops, sports shoe tops, casual shoe tops, brief case, sofa cover, car seat cover, etc., additionally, blazer coat material, glove material, and volleyball material.

Claim of Patent

The fiber base material for artificial leather is coated or immersed in a polymer solution prepared from a water base emulsion and the polymer becomes elastic when bonded, and the polymer solution impregnated in the fiber base material was bonded by wet heat and microwave which is the characteristics of the manufacturing method of artificial leather.

Fig. 1

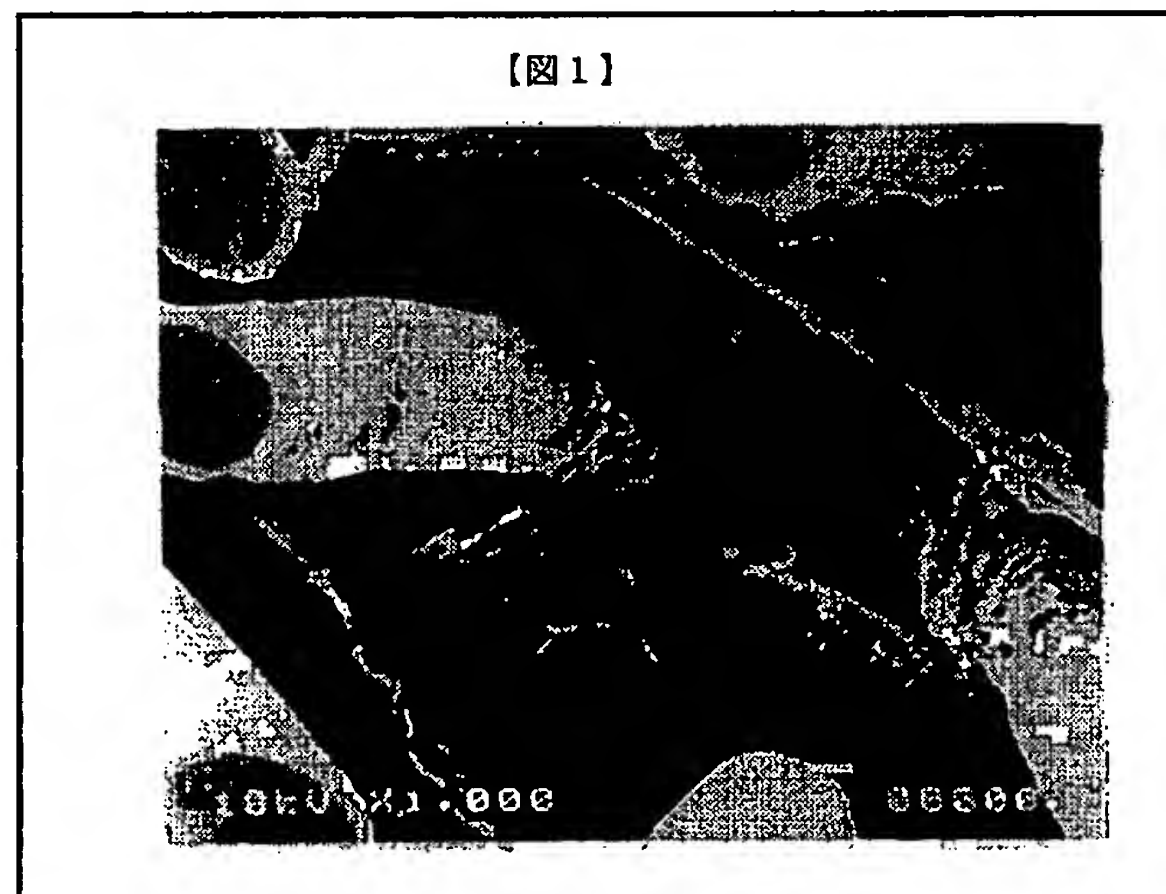


Fig. 2

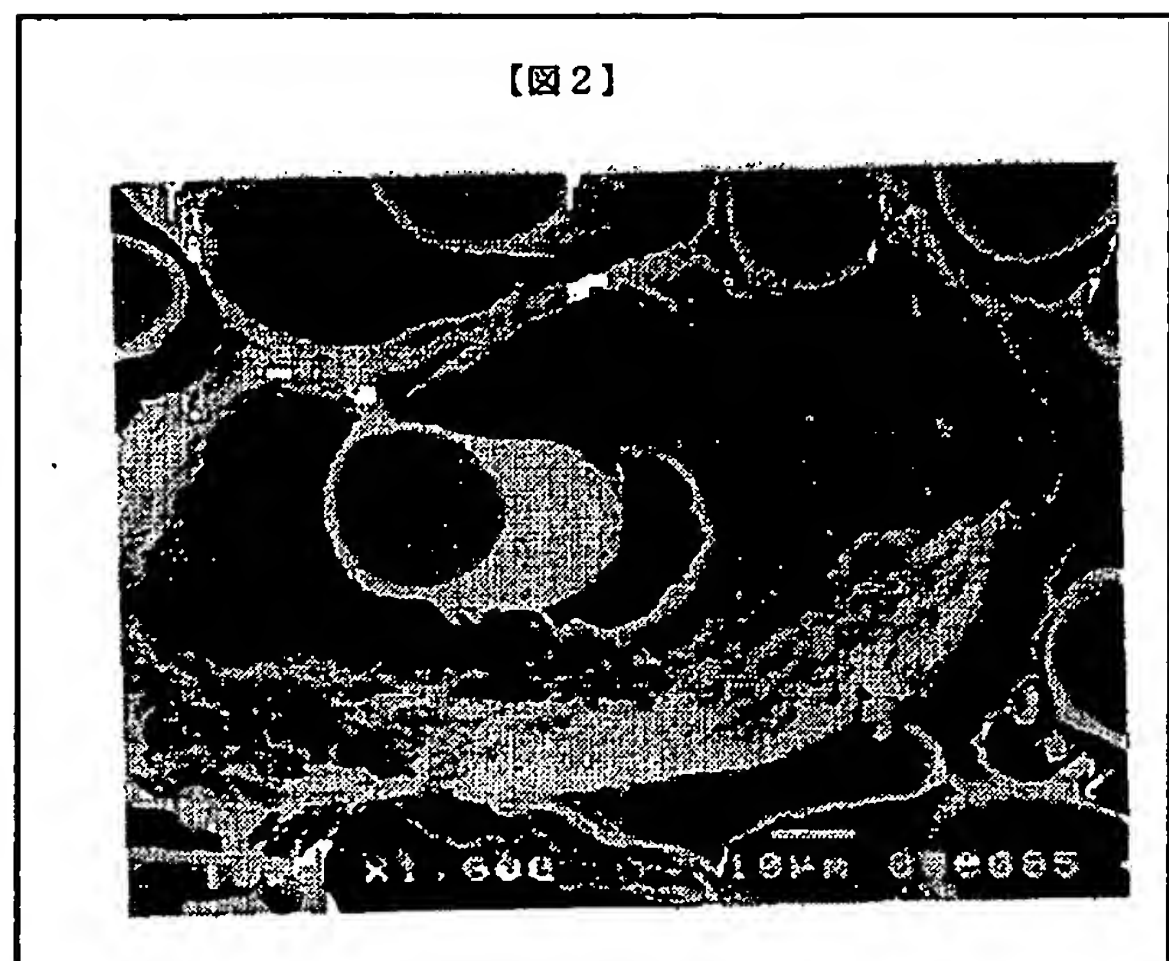


Fig. 3

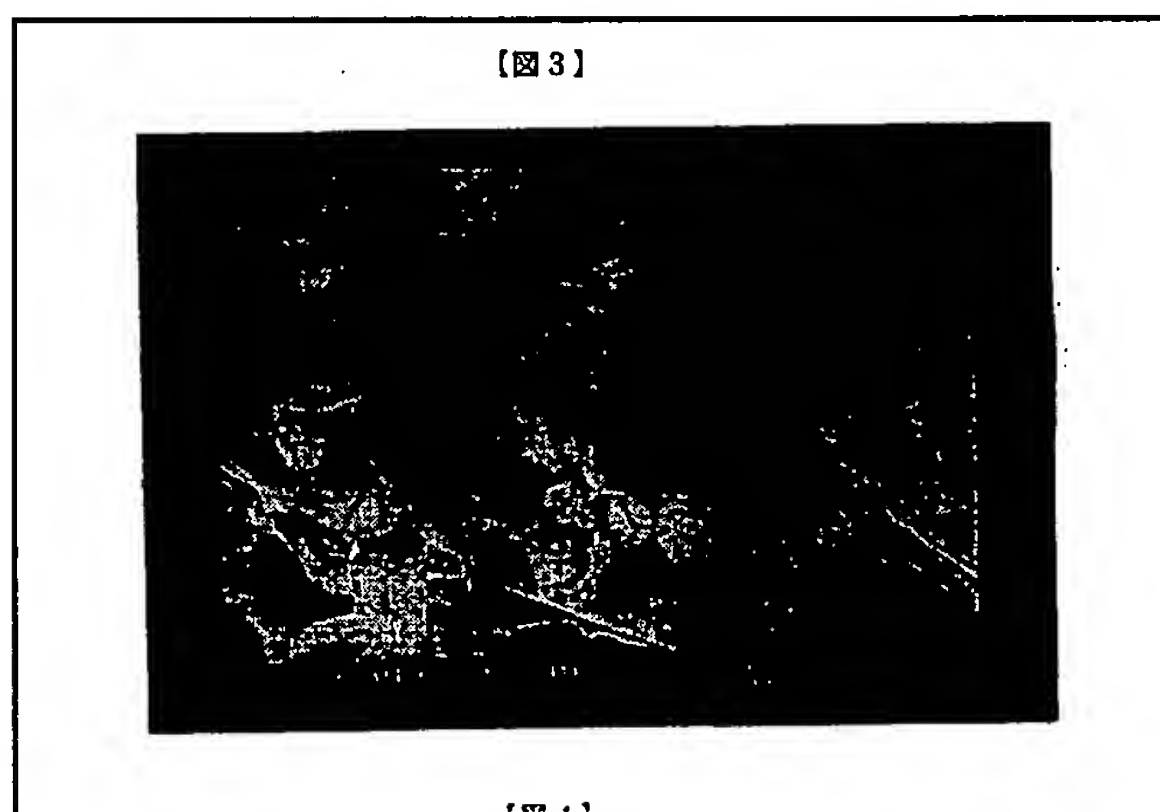


Fig. 4

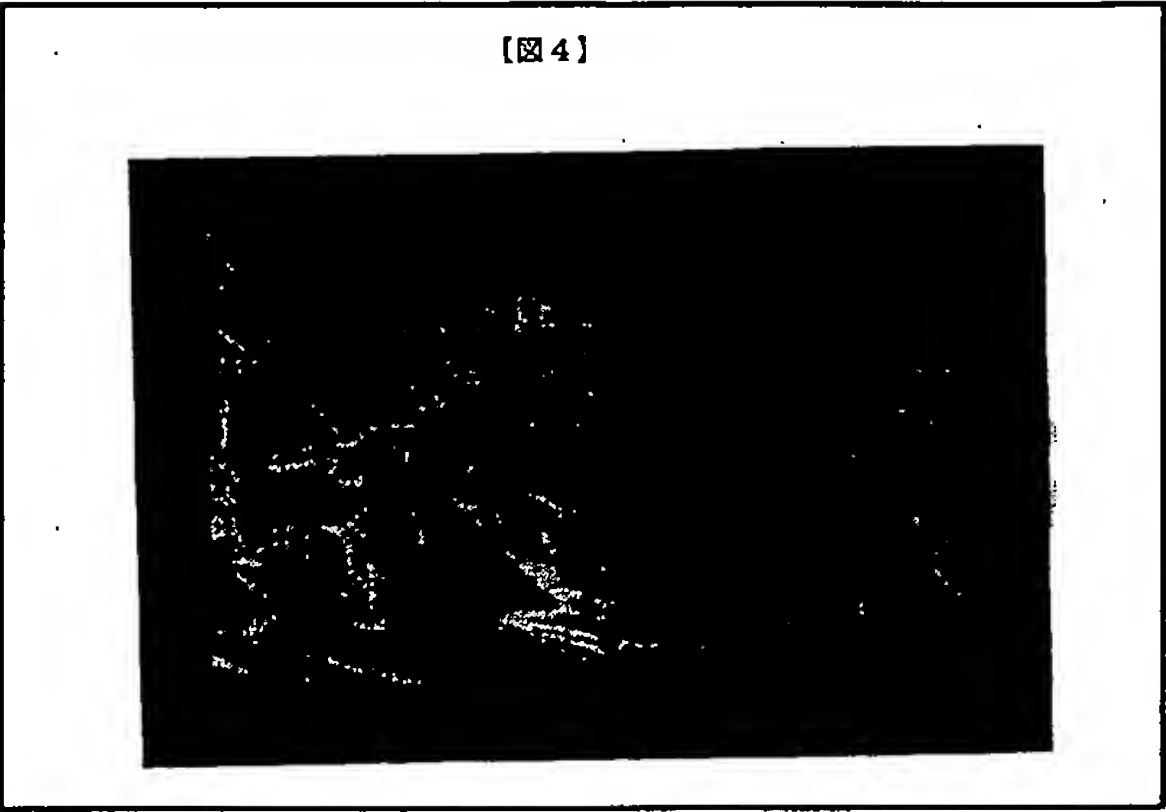


Fig. 5

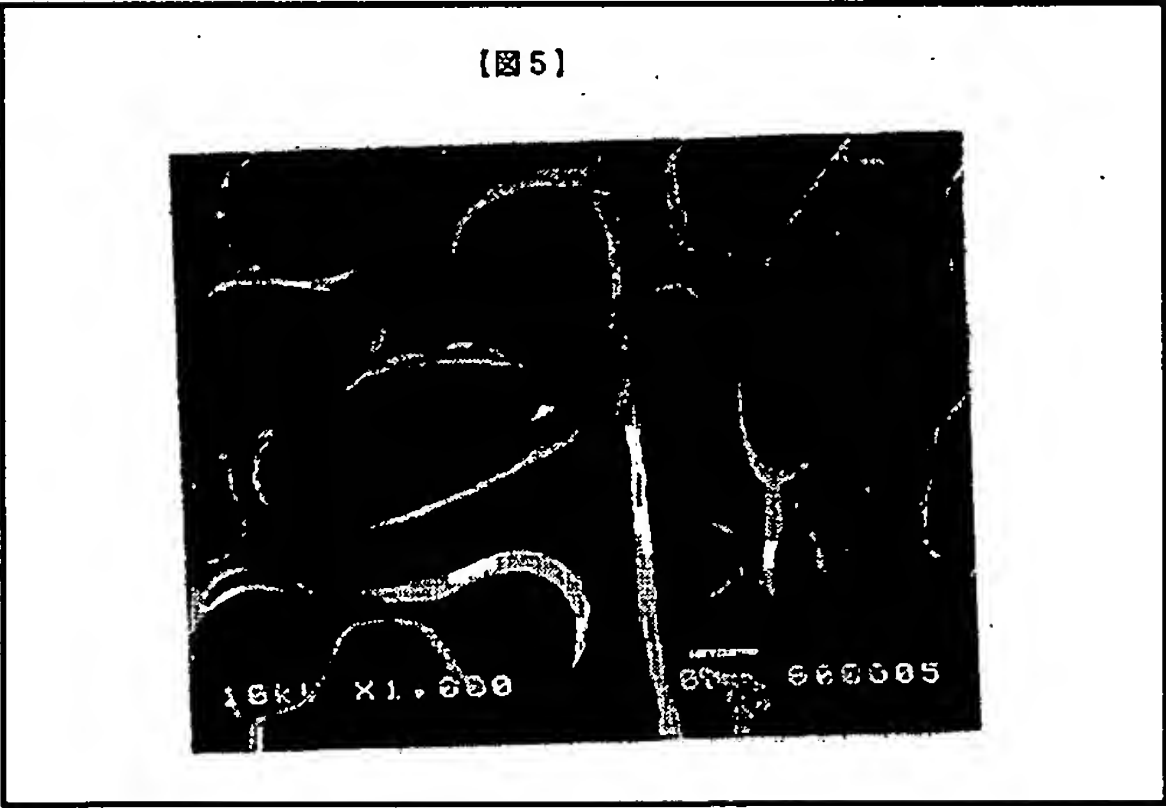


Fig. 6

X-----X

[Base material]-[Immersion coating]-[Wet heat treatment]---[Drying]---

[Product]

Fig. 7

[Fiber base material]-[Shrinking]-[PVA Immersion]-[Drying]-[Coating]-
DMF^m
recycle

(solvent)

-[Bonding]-[Hot water rinse water rinse]-[Squeezing]-[Drying]-
-[Coiling]-[Product]

Fig. 8

[Fiber base material]-[Shrinking]-[Impregnation Coating]-

-[Preheating Far IR heater
[Product]

-[Drying]-[Curing]-

Hot air low temperature drying]